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CRREL

CRREL Research on Materials in Cold Environments

Piyush K. Dutta

December 1990

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Special Report 90-42

U.S. Army Corps of Engineers
Cold Regions Research & Engineering Laboratory

CRREL Research on Materials in Cold Environments

Piyush K. Dutta

December 1990

Prepared for
OFFICE OF THE CHIEF OF ENGINEERS

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PREFACE

This report was prepared by Dr. Piyush K. Dutta, Materials Research Engineer, of the Applied Research Branch, Experimental Engineering Division, U.S. Army Cold Regions Research and Engineering Laboratory. Funding was provided by DA Project 4A762730AT42, *Cold Regions Technology*; Task SS, Work Unit 019, *Behavior of Materials at Low Temperatures*.

The author thanks D. Cole and H. Farquhar of CRREL for technically reviewing this report.

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CRREL Research on Materials in Cold Environments

PIYUSH K. DUTTA

INTRODUCTION

This is the first of a series of reports about the materials research program at CRREL that are expected to become a key element for communication among experts inside and outside of CRREL. They will not replace scientific and technical publications, but will rather survey recent activities of CRREL's materials research program and provide a bibliography covering the period of review.

CRREL will examine cold regions applications of both natural and man-made materials. The range of these materials is quite wide and can include soil (frozen and unfrozen), rock, ice, snow, concrete, metals, plastics, composites, adhesives or explosives. But, because the Army is beginning to extensively use composites in its materiel, the behavior of composites in the cold has received our initial emphasis. Only intermittent studies have been conducted on some dynamic properties of frozen soil and ice; special impedance-matched shock gauges were developed for these studies using a piezopolymer material, polyvinylidene fluoride (PVDF).

The work has progressed in three parallel tasks, with heavier emphasis on the last two. First, a data base of materials' properties at low temperatures was computerized; this is periodically updated through literature searches. It provides a common reference base for most material property data at low temperatures. Second, through a series of tests, low-temperature behavior data for some critical composites intended for military and aerospace applications were generated, and a model to predict composites' low-temperature behavior as lamina in tension was developed. Third, to support the comprehensive program of study of materials' behavior in the cold, essential facilities were set up. This includes not only equipment, testing machines and specialized laboratories, but also developing formal and informal opportunities of exchange among various technical expert groups, both inside and outside of the laboratory. As a multidisciplinary laboratory, CRREL supports materials research from within such relevant areas as fracture mechanics, heat transfer, numerical analysis, chemical analysis and electron microscopy. Outside of CRREL, through its memoranda of understanding, resources are available from the Army Materials Technology Laboratory (AMTL) (Watertown, Massachusetts) and Michigan Technological University (MTU) (Houghton, Michigan). Also, well-known academicians and graduate students from various universities are brought aboard for materials research projects.

The first section of this report, *Program Development*, gives a synopsis of CRREL's materials-property data base and reports important new findings made through studies of the behavior of composites at low temperatures. Results of studies of some special materials (the Navy's glass fiber-reinforced polymeric concrete and the Army's thick polyester–glass composites developed for the Infantry Fighting Vehicle) are discussed briefly. The second part, *Test Facilities Development*, describes the basic test facilities and specialized equipment present at CRREL to comprehensively study materials over a wide range of strain rate loadings and temperatures.

PROGRAM DEVELOPMENT

The materials research program at CRREL takes a long-term view toward serving the Army and the Nation, and has developed through coordination of expertise from the various disciplinary branches within the laboratory, such as civil and geotechnical engineering, geochemical sciences and ice engineering. After its inception in 1985, the program was developed through the three parallel tasks mentioned earlier, including the computerized data base and the materials testing program.

Materials-property data base

We recognized early in the program that computer access to a materials-property data base could be of great benefit to the research and design communities, especially when the data are now widely scattered. Especially for material properties at low temperatures, there were no computerized data bases in the public domain. So, in 1986, CRREL established a low-temperature materials-property data base.

This computerized data base serves as the common reference base at CRREL for most materials-property data. The system has the ability to store, retrieve, analyze, display and manipulate data. To date, about 700 materials and 5000 records obtained from 33 sources have been entered into this data base. Most structural metals, including steel and aluminum, and their alloys, as well as plastics and composites have been covered for temperatures ranging from cryogenic levels to room temperature and above. Numerical data are given for mechanical properties, such as compressive and tensile strengths, impact strength, elastic constants and thermal expansion coefficients. Figure 1 gives a sample page from this data base. Currently, using the dBaseIII software, these data are stored on an IBM PC/AT computer. They are frequently upgraded as new data become available. A manual (Dutta and Healy 1988) helps the users of this data base.

Materials research

Composites

While there are still large gaps in the low-temperature data bases for a wide variety of engineering materials, composite materials have the widest. Composites are being used in increasing quantities in Army materiel because of their favorable strength-to-weight and stiffness-to-weight ratios and their versatility. They have other advantages, such as improved corrosion resis-

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. LIST ALL FOR MATERIAL='STEEL'

Record#	MATERIAL	TEMPERATUR	PROPERTY	METHOD	DATA	UNIT	SOURCE
1914	STEEL ALLOY/HP 9-4-25	80 F	ELONGATION PERCENT	N/A	14.000	N/A	011 P.61
1915	STEEL ALLOY/HP 9-4-25	-100 F	ELONGATION PERCENT	N/A	12.000	N/A	011 P.61
1916	STEEL ALLOY/HP 9-4-25	-200 F	ELONGATION PERCENT	N/A	12.500	N/A	011 P.61
1917	STEEL ALLOY/HP 9-4-25	-320 F	ELONGATION PERCENT	N/A	15.500	N/A	011 P.61
1918	STEEL ALLOY/HP 9-4-25	-423 F	ELONGATION PERCENT	N/A	8.200	N/A	011 P.61
1919	STEEL ALLOY/18 NI MARAGING	80 F	TENSILE STRENGTH	N/A	192.300	KSI	011 P.61
1920	STEEL ALLOY/18 NI MARAGING	-100 F	TENSILE STRENGTH	N/A	217.600	KSI	011 P.61
1921	STEEL ALLOY/18 NI MARAGING	-200 F	TENSILE STRENGTH	N/A	234.300	KSI	011 P.61
1922	STEEL ALLOY/18 NI MARAGING	-320 F	TENSILE STRENGTH	N/A	268.000	KSI	011 P.61
1923	STEEL ALLOY/18 NI MARAGING	-423 F	TENSILE STRENGTH	N/A	313.000	KSI	011 P.61
1924	STEEL ALLOY/18 NI MARAGING	80 F	ELONGATION PERCENT	N/A	7.000	N/A	011 P.61
1925	STEEL ALLOY/18 NI MARAGING	-100 F	ELONGATION PERCENT	N/A	6.000	N/A	011 P.61
1926	STEEL ALLOY/18 NI MARAGING	-200 F	ELONGATION PERCENT	N/A	6.300	N/A	011 P.61
1927	STEEL ALLOY/18 NI MARAGING	-320 F	ELONGATION PERCENT	N/A	8.000	N/A	011 P.61
1928	STEEL ALLOY/18 NI MARAGING	-423 F	ELONGATION PERCENT	N/A	8.200	N/A	011 P.61
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1933	STEEL ALLOY/18 NI MARAGING	-423 F	TENSILE STRENGTH	N/A	244.400	KSI	011 P.61
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2083	STEEL STAINLESS AISI 304	-200 F	TENSILE STRENGTH	N/A	209000.000	PSI	015 P.218
2084	STEEL STAINLESS AISI 304	-100 F	TENSILE STRENGTH	N/A	175000.000	PSI	015 P.218
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2086	STEEL STAINLESS AISI 304	70 F	TENSILE STRENGTH	N/A	106000.000	PSI	015 P.218
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2090	STEEL STAINLESS AISI 304	0 F	ELONGATION PERCENT	N/A	50.000	N/A	015 P.221
2091	STEEL STAINLESS AISI 304	70 F	ELONGATION PERCENT	N/A	62.000	N/A	015 P.221
2092	STEEL STAINLESS AISI 304	-300 F	IMPACT STRENGTH	N/A	67.000	FT-LB/IN	015 P.232
2093	STEEL STAINLESS AISI 304	-200 F	IMPACT STRENGTH	N/A	68.000	FT-LB/IN	015 P.232
2094	STEEL STAINLESS AISI 304	-100 F	IMPACT STRENGTH	N/A	68.500	FT-LB/IN	015 P.232
2095	STEEL STAINLESS AISI 304	0 F	IMPACT STRENGTH	N/A	69.500	FT-LB/IN	015 P.232
2096	STEEL STAINLESS AISI 304	70 F	IMPACT STRENGTH	N/A	70.000	FT-LB/IN	015 P.232

. USE REFERENCE

011 P.61 MARTIN, H.L., MILLER, P.C., INGRAM, A.G. AND CAMPBELL, J.E. (1968) EFFECTS OF LOW TEMPERATURES ON THE MECHANICAL PROPERTIES OF STRUCTURAL METALS. NATIONAL AERONAUTICS AND SPACE ADMINISTRATION, WASHINGTON, D.C.

015 P.221 SCHWARTZBERG, F.R., OSGOOD, S.H., KEYS, R.D. AND KIEFER, T.F. (1964) CRYOGENIC MATERIALS DATA HANDBOOK. AIR FORCE MATERIALS LABORATORY, WRIGHT-PATTERSON AIR FORCE BASE, OHIO.

Figure 1. Sample page of the materials-property data base.

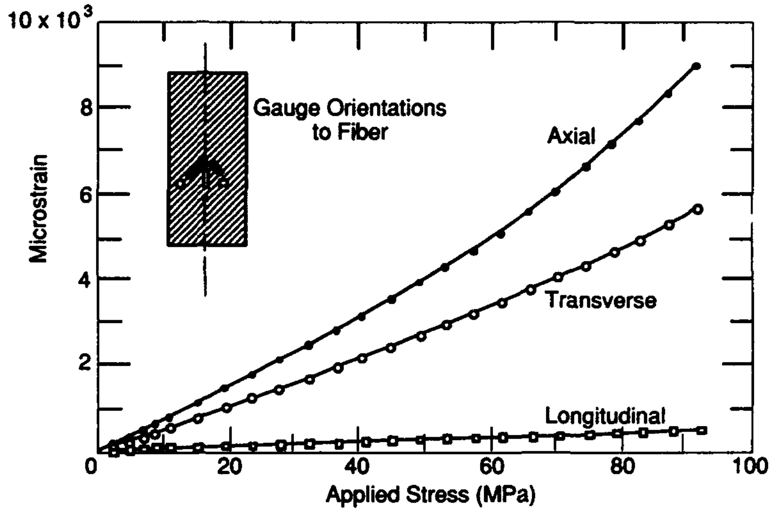
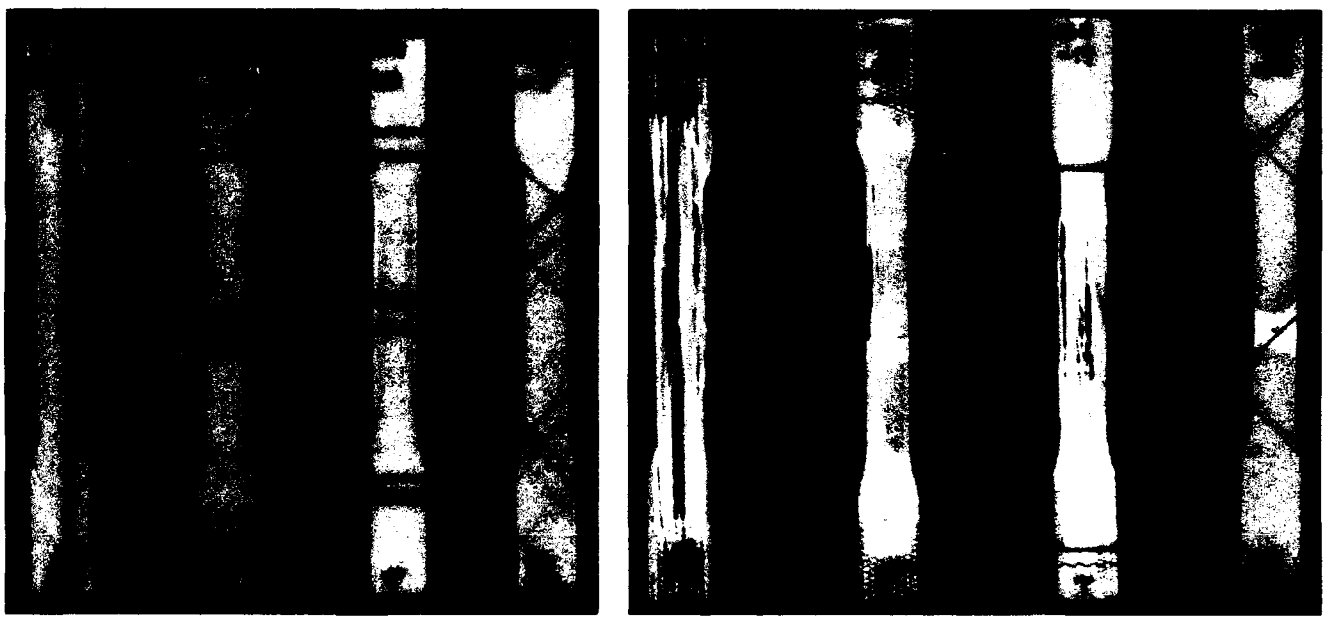


Figure 2. Anisotropic stress distribution data from three pairs of strain gauges on a unidirectional laminate.

tance, improved crash safety and vibration suppression. In military applications, additional advantages are derived from lower electromagnetic signatures and superior thermal insulation.

Composites are formulated by suitably mixing two or more compatible components—usually a matrix and a reinforcing material. The list of candidate materials for matrices and reinforcements is almost limitless, and the rate of development of new composites is almost dizzying. Under different environmental conditions, their responses vary. Low temperature degrades their durability because of mismatches between the components' thermal expansion coefficients. It also makes them brittle, which influences the composite's impact resistance. It is essential to know the significance of these problems in cold regions applications.

To characterize the influence of the cold, we focused our attention on the effects of low temperature and low-temperature thermal cycling on the tensile strength of a number of composite materials. Approximately 300 specimens have been tested, the majority of which were instrumented with up to six strain gauges. A computerized data acquisition system monitored the gauges and the applied load from which elastic moduli and Poisson's ratio were determined. Figure 2 gives a typical plot of stress–strain data from three pairs of strain gauges mounted on a $[\pm 45]_6$ graphite–epoxy laminate. Some of these materials have been tested at temperatures as low



a. Before. *b. After.*

Figure 3. Glass fiber laminates before and after tensile tests.

as –60°C and some were thermally cycled to the lowest temperature of –180°C.

Figure 3 shows four types of glass fiber–epoxy laminate composites before and after testing in tension at low temperature. Figure 4 shows results over a range from room temperature (23°C) down to –40°C, indicating that the ultimate tensile strength of uniaxial laminates ($[0]_6$) decreases at lower temperatures. Longitudinal fiber-dominated samples ($[90_2 0_2]_s$) also showed a slight decrease in strength. The strength of laminates with a lower proportion of 0° oriented fibers ($[90_2 0]_s$) actually increased, but off-axis specimens $[45/\text{-}45]_6$ showed no change in strength.

Two different manufacturer's uniaxial graphite–epoxy composites (Fiberite and Hercules) were studied to characterize their low-temperature behavior. Figure 5 shows tensile test data for their longitudinal fiber direction. Like the uniaxial laminates of glass fiber–epoxy,

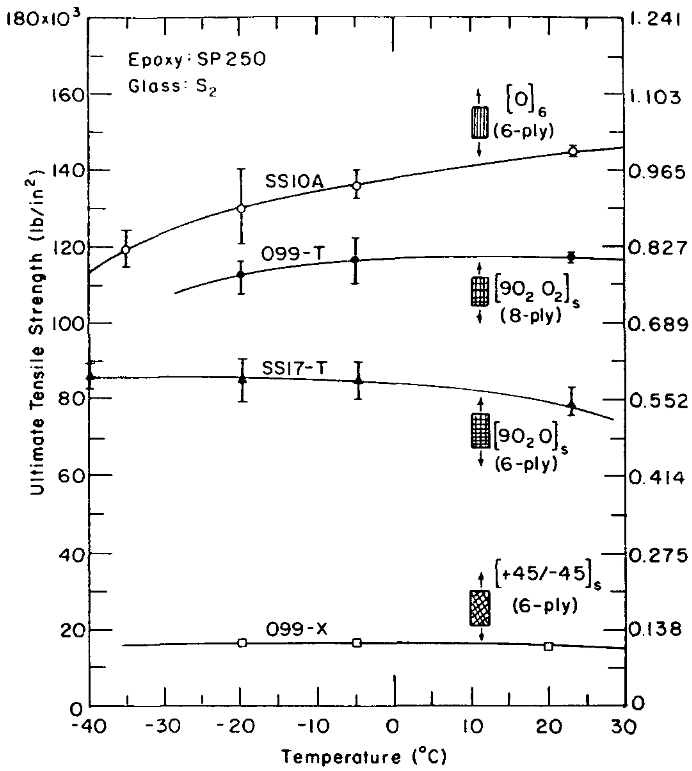


Figure 4. Influence of temperature on tensile strength of glass fiber–epoxy laminate.

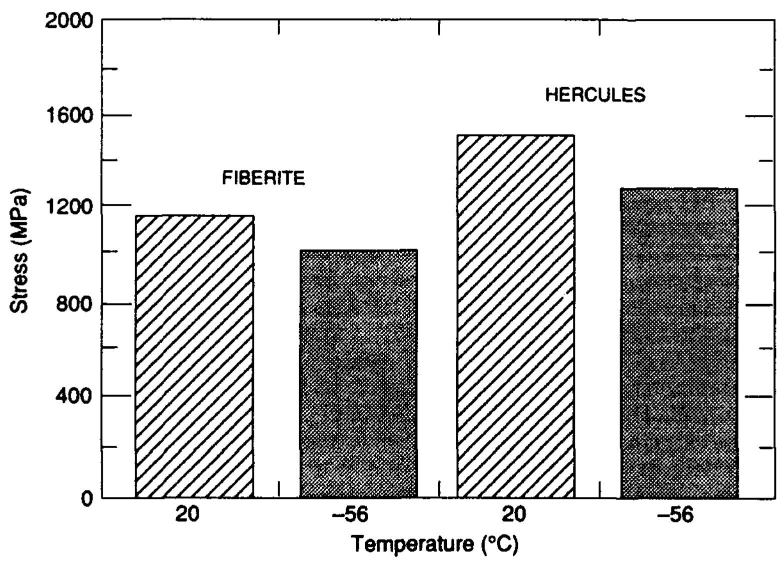


Figure 5. Influence of temperature on tensile strength of graphite–epoxy laminate.

Table 1. Comparison of room temperature and low temperature tensile strength data of unidirectional laminates.

	Laminates	Room temperature data (°C)	Room temperature data (Mpa)	Low temperature data (°C)	Low temperature data (Mpa)	Percentage reduction	Reference
A. Graphite epoxy							
(1)	T300/5208 dry	22.2	1358	–53.9	1193	12.1	Springer (1984)
	$[0]_6$ wet	22.2	1641	–53.9	1510	8.0	
(2)	Celion 6000/PMR 15 $[0]_8$	23.8	1448	–156.7	1524	–5.2	Garber et al. (1980)
(3)	P75-S/CE 3	23.8	793	–128.9	414	47.8	Mazzio and Huber (1984)
	$[0]_5$	23.8	793	–184.4	345	56.5	
(4)	T300/Fiberite 934	23.8	1600	–128.9	1276	20.3	Mazzio and Huber (1984)
	$[0]_?$	23.8	1600	–184.4	1103	31.0	
(5)	PRD49-III/FRLB 4617	23.8	2206	–198.6	1655	25.0	Hanson (1972)
	$[0]_?$	23.8	2206	–252.8	1793	18.8	
(6)	2002/HMS graphite and 2002/S901 glass hybrid	25.0	896	–162.2	552	36.9	Philpot and Randolph (1982)
(7)	T300/914C $[0]16$	20.0	1772	–60.0	1627	8.2	DFVLR (1985)
(8)	T300/Code 69 $[0]_{16}$	20.0	1496	–60.0	1269	15.2	DFVLR (1985)
(9)	T300/F550 $[0]_{16}$	20.0	1593	–60.0	1351	15.2	DFVLR (1985)
(10)	T40/974 $[0]_7$	23.8	1186	–56.1	1034	12.8	Dutta et al. (1988)
(11)	AS-4/3501 $[0]_7$	23.8	1662	–56.1	1441	13.3	Dutta et al. (1988)
B. Fiberglass–epoxy							
(1)	5-2/SP250 $[0]_6$	23.8	993	–35.0	827	16.7	Dutta et al. (1988)
C. Kevlar							
(1)	Kevlar/J2	23.8	1103	–50.0	972	11.9	Dutta (1989)
D. Boron/epoxy							
(1)	NARMCO 5505 Boron/epoxy	23.8	1434	–55.0	1386	3.3	Dept. of Defense (1973)
(2)	SP272 Boron/epoxy	23.8	1296	–55.0	1269	2.1	Dept. of Defense (1973)

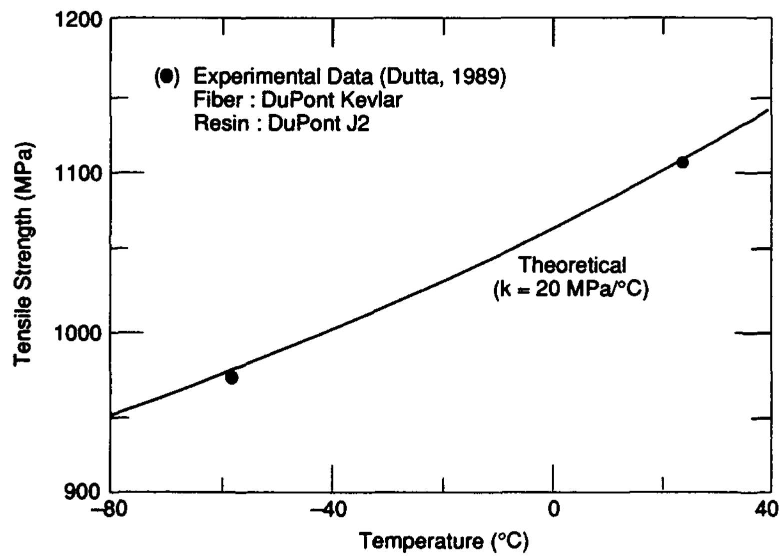


Figure 6. Influence of low temperature on tensile strength of Kevlar/DuPont J-2 resin laminate.

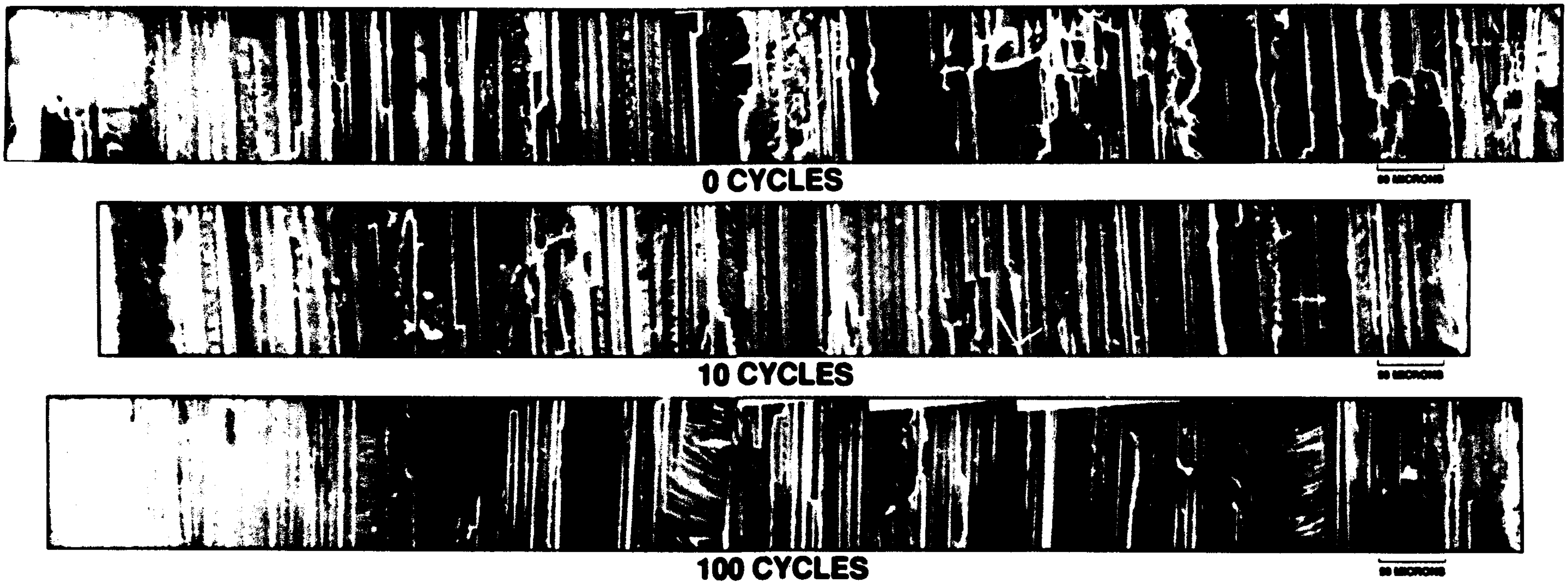


Figure 7. Graphite epoxy fracture surface micrograph showing progressive smoothing of the surface with thermal cycling.

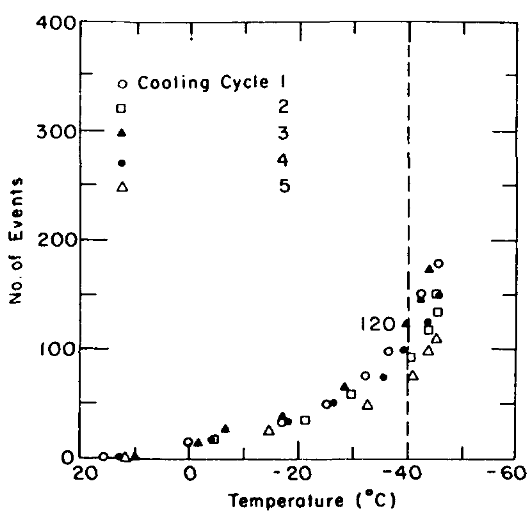
these two uniaxial laminates also show a decrease in tensile strength values at low temperature. We also observed reduced tensile strength at low temperature in Kevlar/DuPont J2-resin (Fig. 6). Many other data (Table 1), scattered throughout the composite-materials literature, have also shown low-temperature strength degradation for the uniaxial composites. An explanation of such behavior, based on stiffening of the resin at low temperature and the waviness of fine fibers, has recently been developed (Dutta 1989).

Both glass fiber–epoxy and graphite–epoxy laminates were systematically tested to study their durability under low-temperature thermal cycling. The strength of unidirectional laminates of graphite–epoxy composites that were subjected to extremely low-temperature (−180°C) thermal cycling increased in the longitudinal direction but decreased in transverse (90°) and off-axis (45°) directions. The thermal cycling induced microcracks in the matrix, weakened and softened it, and, therefore, decreased the strength in the matrix-dominated transverse and off-axis directions. The transverse fracture surfaces, examined under the Scanning Electron Microscope (SEM) (Fig. 7), showed a progressive increase in the smoothness of the surface with increasing thermal cycling; the increased smoothness of the surface is associated with lower strength. In the longitudinal loading direction, the softer matrix allowed the wavy fibers to realign, providing a more uniform load distribution, which resulted in a higher load before failure.

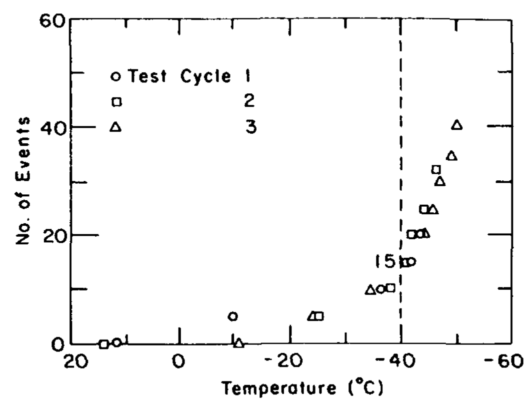
Analyzing low-temperature stresses in composite laminates having fibers in each ply oriented in different directions is a relatively more complex problem. In this case, the thermal expansion coefficient mismatch must be considered, not only between the components of the matrix and fibers, but also between each layers (plies). In each layer, the thermal expansion coefficient is anisotropic, usually having its maximum value in the direction transverse to the fiber and minimum along the fiber, so a change in temperature induces inter-laminar shear stress. Large temperature excursions can develop sufficient stress to cause microcracks or flaws in the material. Using the acoustic emission technique, we studied the growth of microcracks with decreasing temperature for a number of glass fiber–epoxy laminates. The results (Fig. 8) show evidence of more microcrack growth in multidirectional-ply laminates than in unidirectional-ply laminates. Microcrack growth from prolonged thermal cycling will degrade a laminate's strength. Figure 9 illustrates the progressive degradation of tensile strength of a six-ply glass fiber–epoxy laminate following low-temperature thermal cycling.

These studies show that the fundamental problem of composites at low temperature is the development of internal stresses from the differential contraction and expansion of the components, which promote the growth of microcracks. Designs that incorporate composites having large differences in internal thermal expansion coefficients *must* consider the influences of the cold on performance.

Additional studies conducted on a polyester–glass composite show a drastic change in the compressive properties of the material. Figure 10 gives the results of testing 38-mm-diameter cylindrical specimens under compression at room temperature and at −48°C. The

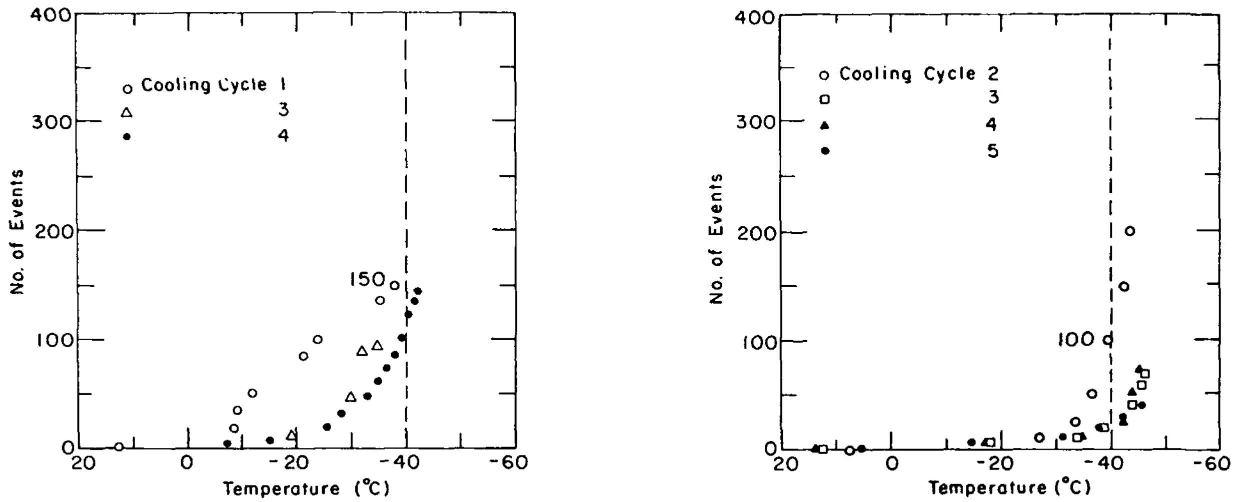


a. The $[90_2 0_2]_s$ eight-ply glass fiber (S-2)–SP250 resin.



b. The $[0]_6$ six-ply glass fiber–SP250 resin.

Figure 8. Acoustic emission data from cooling laminates of different ply orientations.



c. The $[+45_2/-45]_s$ six-ply glassfiber (S-2)–SP250 resin.

d. The $[90_2 0_2]_s$ six-ply glass fiber (S-2)–SP250 resin.

Figure 8 (cont'd). Acoustic emission data from cooling laminates of different ply orientations.

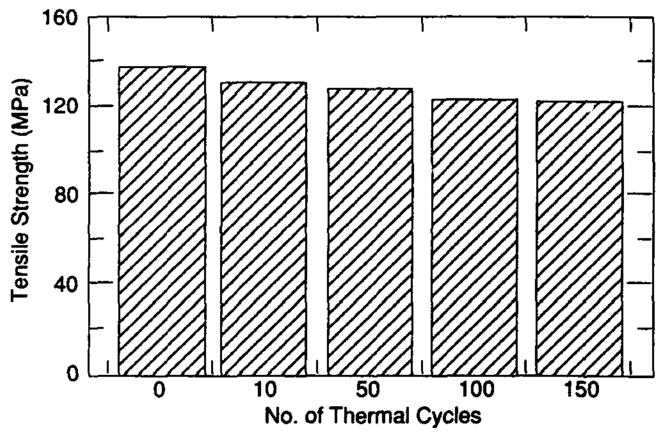


Figure 9. Progressive strength degradation of glass fiber–epoxy laminate from low-temperature thermal cycling.

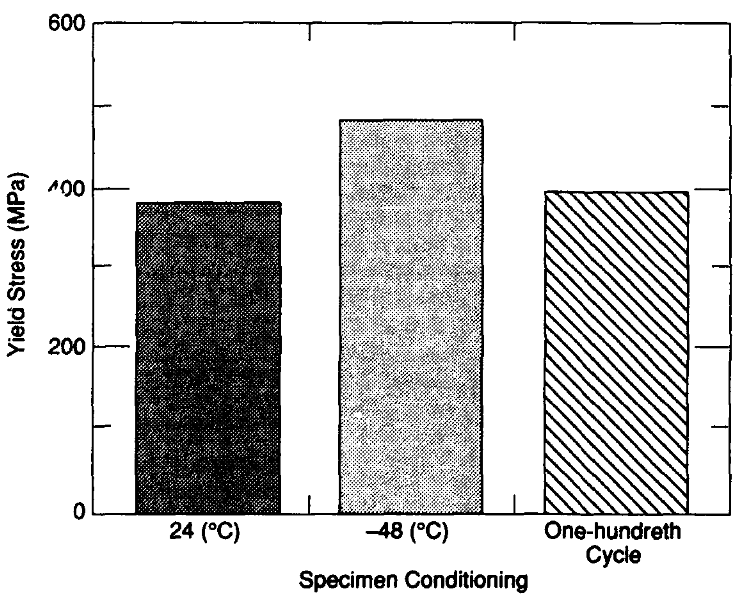


Figure 10. Compressive strength increase of glass–polyester composites at low temperature.

Before — *Room temperature 24°C* — *Low temperature −48°C*

Figure 11. Catastrophic brittle failure of glass–polyester composites at low temperature.

compressive strength increased by 17.6% at low temperatures, but the specimen failed more violently (Fig. 11), owing to ductile-to-brittle transition. The energy absorption before failure at low temperature is higher than it was at room temperature. In designing structures where crushworthiness is an important consideration, for example in automotive structures, the low-temperature induced energy release rate must be carefully considered.

Special materials

The influence of low temperatures on Young's modulus and shear modulus for thick composites developed for the Infantry Fighting Vehicle were studied. These composites (Fig. 12) were specially formulated with plain weave S-2 glass reinforcement in polyester resin, with about 53% volume fraction of fibers. The tests involved flexing three different sizes of beams of this material and measuring deflections and loads accu-

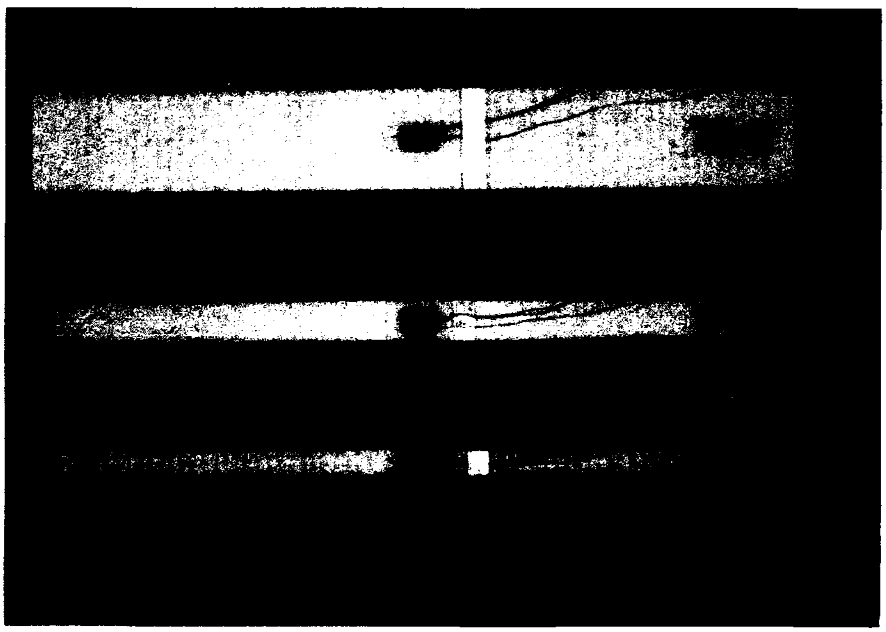


Figure 12. Special test specimens of a thick polyester–S2-glass composite for elastic moduli testing by flexure.

Test	Dimensions (in.)	No. of Specimens	Specimens
Compression	1-1/2 x 3/4 x 5/16	A 15 B 13	
Tension	4-1/2 x 3/4 x 5/16	A 10 B 10	
Charpy impact	1-1/2 x 1/2 5/16	A 16 B 18	
Hopkinson bar impact	1-1/2 D x 5/16	A 12 B 12	

Figure 13. Test specimens of U.S. Navy's glass fiber reinforced polymer concrete.

rately using an MTS load frame equipped with a temperature-controlled cold chamber. Test temperatures were set at three different points, 27, –40 and –57°C, at each of which all three sizes of specimens were tested. The data were analyzed using the procedures developed by Fischer et al. (1981) for simultaneous determination of both Young's modulus and shear modulus at each of those temperatures. The room temperature modulus values compared well with those determined at the AMTL. The modulus values from tests at –40 and –57°C were also not significantly different from the room temperature values.

At the request of the U.S. Navy, the cold weather durability of another special material, a proprietary Glass-Fiber Reinforced Polymer Concrete (GFRPC) was studied. Two formulations of this material were to be evaluated for applications in near-shore structures. Degradation from salt water and reduction of impact resistance at low temperatures, especially after several years of cold weather cycling, were the main concerns. After saturating them with salt water, we cold cycled representative samples of these materials in the laboratory and tested them at low temperature (–20°C) for compression, tension, Charpy impact and high-strain-rate Hopkinson bar impact strengths (Fig. 13). These data were then compared with the base line data obtained from the untreated specimens tested at room temperature (27°C). There was no degradation of impact resistance; on the other hand, as a result of low temperature and thermal cycling, both compressive and tensile strengths improved.

TEST FACILITIES

The fundamental mechanical response of structural materials is greatly influenced by two factors: temperature and rate of loading. Although not interrelated in applications, the influence that they have on the material is almost alike. For example, both low temperatures and high rate loadings make many materials brittle. Therefore, a comprehensive program to study impact of cold on materials must also include the loading rate effects. Our testing facilities were developed for examining not only the low-temperature behavior of materials, but also for doing high-load-rate tests at those temperatures. A brief review of these facilities now follows.

Universal testing machines

There are several servo-controlled universal testing machines at CRREL, of which the 1.112-MN capacity, controlled environment (to –60°C) MTS machine (Fig. 14) is the main workhorse. Installed in 1971, this machine has been used for mechanical testing of innumerable samples of ice, rock, frozen and unfrozen soil, concrete, composites and other materials. Fixtures and facilities have been developed for uniaxial, biaxial and triaxial as well as flexural testing of materials. The control console, MTS model 445, provides for maximum platen displacement of 150 mm at a maximum rate of 42 cm/s. Tension–tension, compression–compression or tension–compression fatigue tests can be performed at the maximum cycle rate of 10 per second. The environment chamber is cooled by use of a thermostatically controlled refrigeration system that circulates cold air through the chamber. Integrated into the system are several data acquisition devices, which for convenience include an X–Y plotter, a chart recorder and an eight-channel MEGADAC model 2200C datalogger. In conjunction with the last, a desk-top PC can acquire, manipulate, store or, on demand, print out numerical and graphical data using commercially available software. Adjacent to the testing machine, a dedicated coldroom, capable of operating down to –20°C, facilitates preparation of ice, frozen soil or other similar cold samples. The system is calibrated every year.

The intermediate capacity (245-kN) MTS universal testing machine has been installed in the CRREL Mate-

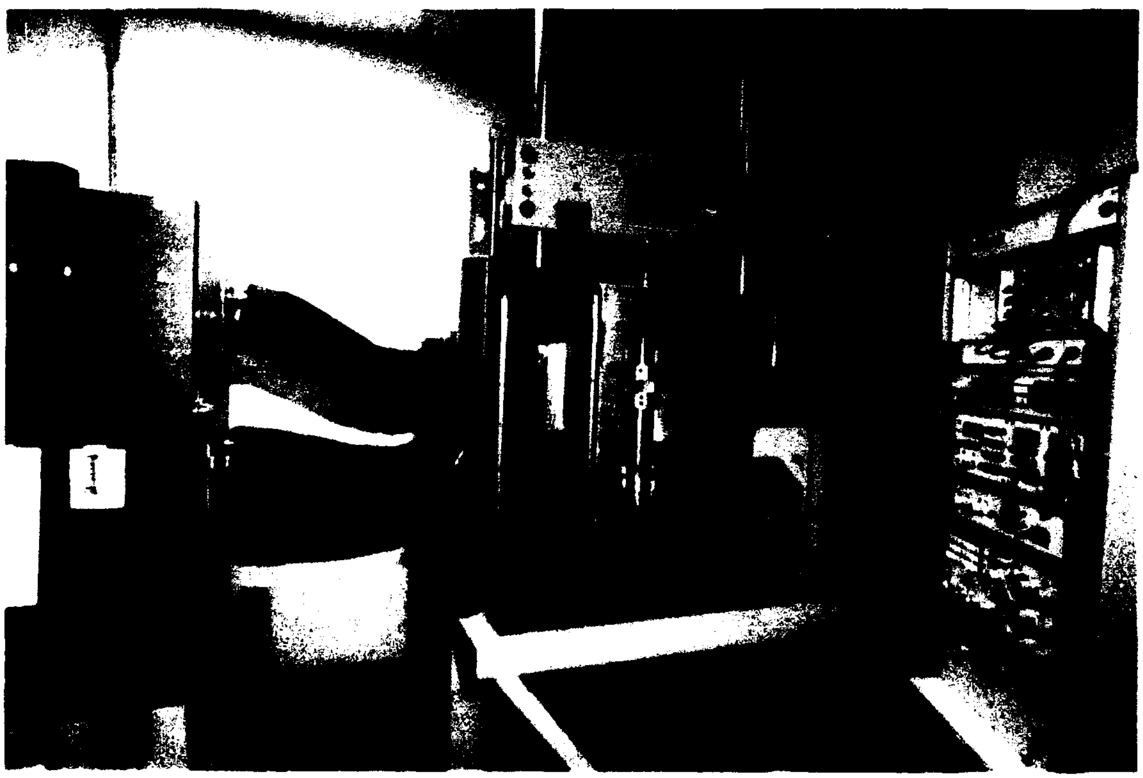


Figure 14. High-capacity (1.112-MN) servo-hydraulic testing machine for low-temperature testing.

rials Engineering Research Laboratory primarily for testing of man-made materials. Liquid nitrogen will be used for cooling its environmental chamber down to –100°C. Like the high capacity machine, this machine is also computer-controlled, and a computer-based data acquisition system has been installed. Its maximum platen displacement is 150 mm; on demand, its servo-control system can control the platen motion for a constant-stress-rate test.

For low range loading tests, another MTS machine of 111-kN capacity is available. Installed in the Soils Testing Laboratory, this machine has a finely tuned thermostatically controlled environmental chamber with a Freon refrigeration system, which can allow testing at temperatures as low as –40°C. The controller console has a ramp output that provides a maximum 150-mm platen displacement at the fastest speed of 42 cm/s. Chart recorders, X–Y plotters or computers can easily be interfaced with this machine for automatic data acquisition.

For precision testing and measurements at very light loads, as is normally required for many plastics, composites and adhesives, a bench-top Tinius Olsen model 1000 machine has been installed. This 5000-N capacity machine can provide a maximum of 735 mm of travel at the fastest speed of 8.3 mm/s. It will accept a small environmental chamber that is cooled by liquid nitrogen for low-temperature testing.

There are other mechanical testing machines available at CRREL of various load capacities. A Tinius Olsen 88-kN machine, installed permanently in a coldroom with control consoles located outside, is available for long-term low-temperature testing (e.g., creep tests). Another high-capacity (1.334-MN) room-temperature machine, installed in the Soils Testing Laboratory, allows large size concrete, rock or soil samples to be studied.

Charpy impact testing machine

CRREL's Charpy impact testing machine (Fig. 15) has been installed in the Materials Engineering Research Laboratory according to procedure AMXMR-P-702-104, recommended by AMTL (1969). The Charpy test assesses a material's ability to withstand brittle fracture. For many materials, especially steel, it is essential to know this property at low temperatures, where their brittleness increases drastically. In the machine, the test specimen, after being soaked at the specified temperature, is fractured by the impact of a swinging pendulum. The pendulum's energy before and after it breaks the specimen gives the data to determine the material's brittle fracture susceptibility. ASTM (1989) standard E-23 type tests are conducted using this machine. The CRREL machine, a SATEC model SI-1D3, covers a range of 34 to 407 J of energy in six different settings.

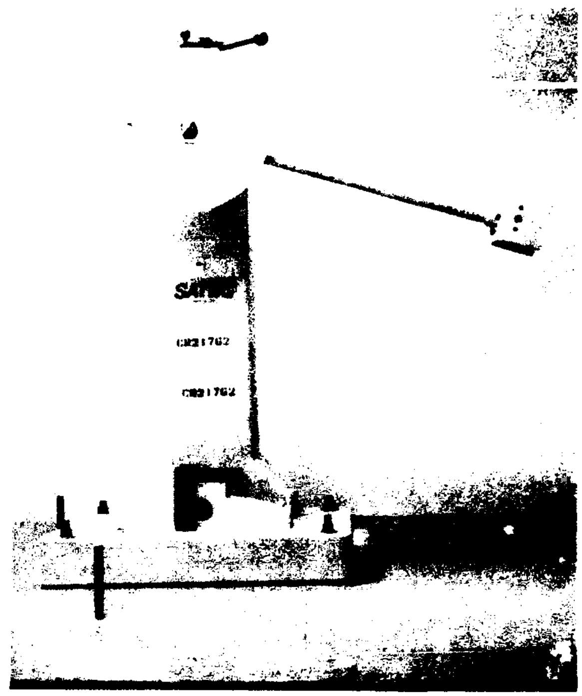


Figure 15. Charpy impact test machine.

Drop shock tester

An L.A.B. model SD-24 free fall shock machine (Fig. 16) installed in the Materials Engineering Research Laboratory provides shock evaluation of specimens to meet military (Mil-Spec) or industrial (ASTM) specifications. This is also an important research tool for its capability to generate geometrical shock pulses (half-sine, sawtooth or square) for energy delivery at a controlled rate. The 245-kg drop carriage falling from 152.4 cm gives a 1000-g maximum acceleration in a minimum 1-ms-duration pulse. Critical shock waveform parameters, including peak gravities, pulse duration, impact velocity and velocity change, are digitally calculated and displayed on a four-channel Nicolet model 4094 mainframe and two 4562 plug-ins with 12-bit resolution. Digital data from the Nicolet oscilloscope are transferred to the desk-top PC for calculation of energy expended and other analyses. Like the Charpy test, both cold and room-temperature specimens can be impact tested in this machine.

Hopkinson pressure bar apparatus

While the universal testing machines, the Charpy and the drop tester provide an increasing rate-of-strain loading, the maximum is around 10 strains per second (Farrell and Dutta 1986). A still higher rate-of-strain loading is possible in the Hopkinson Pressure Bar Apparatus (HPBA). The HPBA, installed in the Materials Engineering Research Laboratory (Fig. 17), is suitable

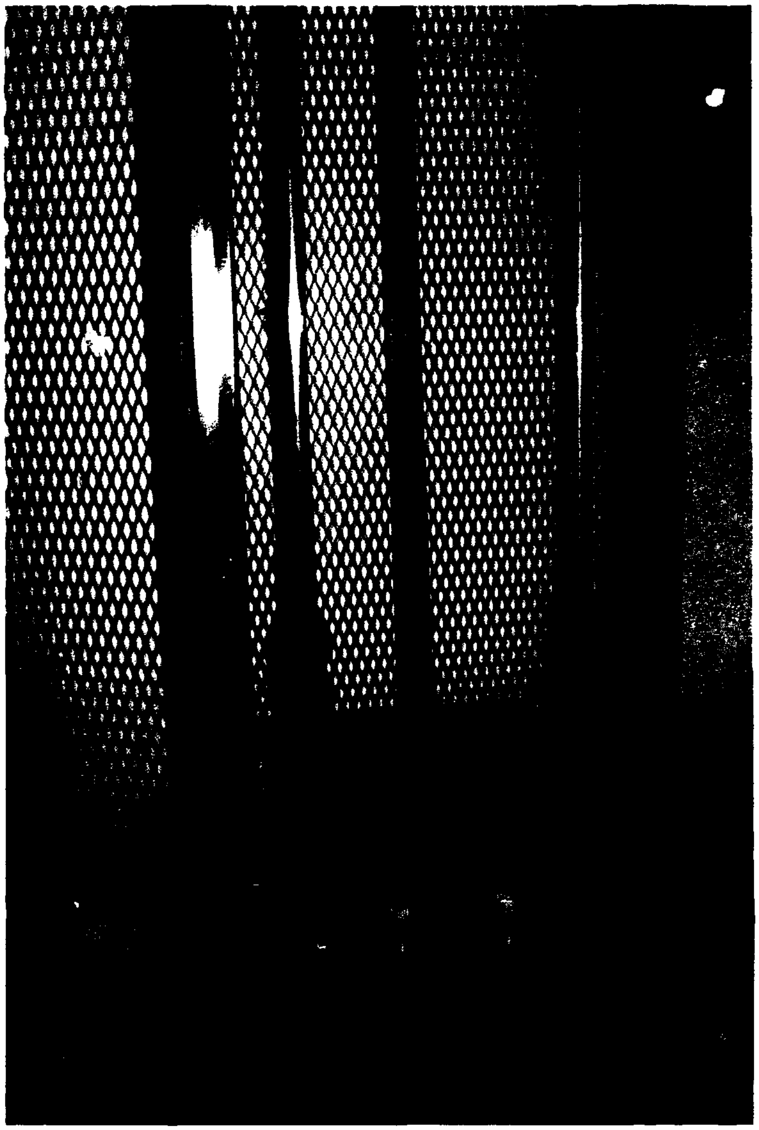
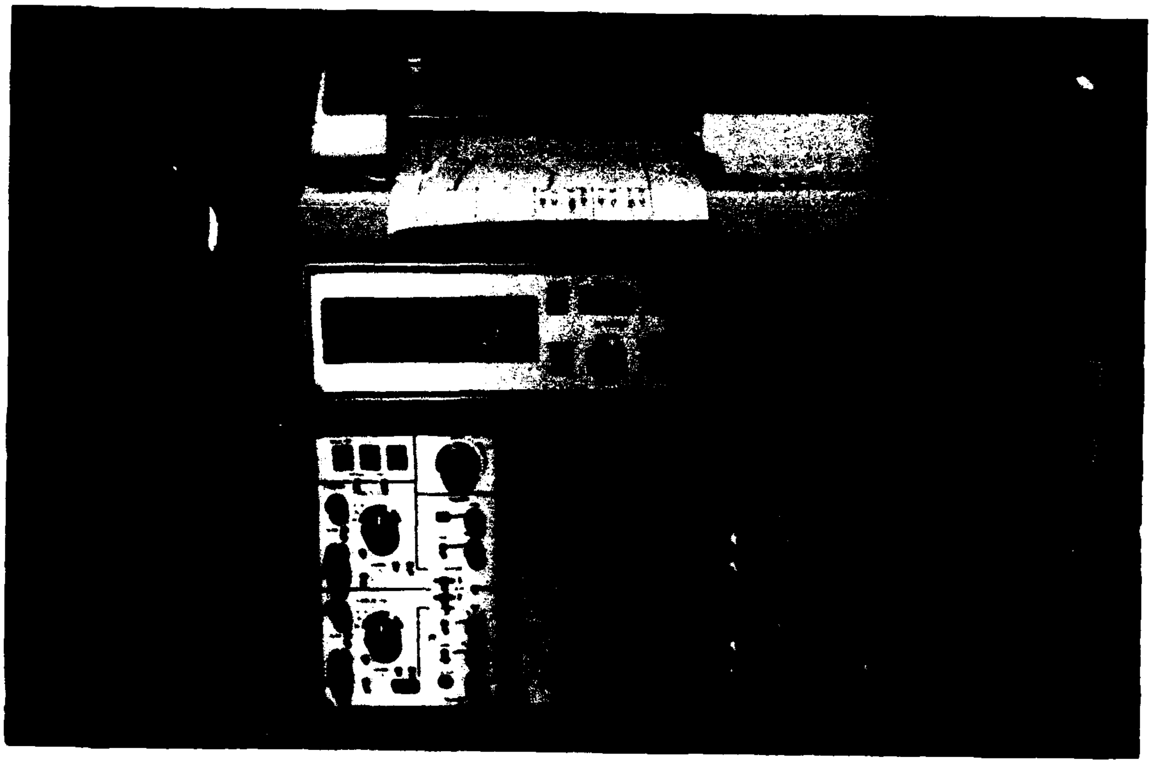


Figure 16. Free fall shock testing machine.

Figure 17. Hopkinson pressure bar test system for high-strain-rate measurements at low temperatures.



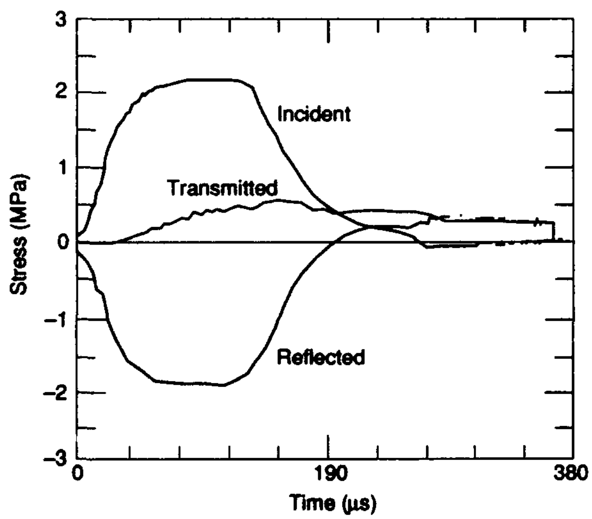
a. Dynamic stress–strain relationship for an ice sample computed from the stress pulse waveform.

b. Incident, reflected and transmitted stress pulse waves reconstituted on the computer screen.

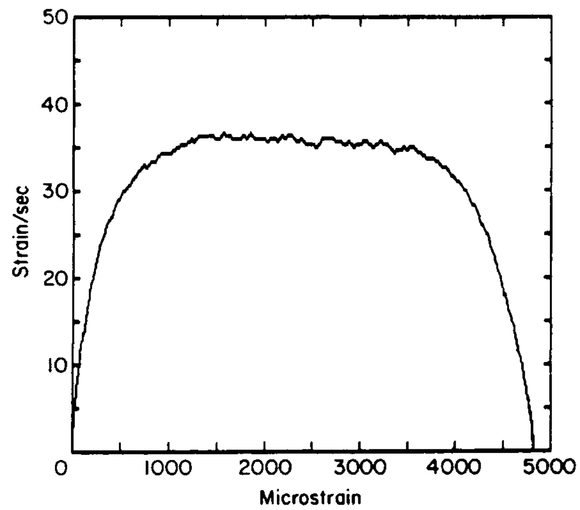
Figure 18. Real-time data acquisition and analysis system for Hopkinson bar apparatus.

for deformation tests on natural materials at 10 to 100 strains per second. High impedance man-made materials can be tested at even higher rates. The apparatus consists of a short striking bar driven by compressed air and two long, solid, round bars instrumented with strain gauges. The striker's impact on the first bar sets up a compressive stress pulse, whose amplitude depends on the impact velocity and whose duration depends on the striker's length. The test specimen is held between the two bars in a chamber cooled by liquid nitrogen. At the interface, some energy of the stress pulse from the first bar is reflected because of the mechanical impedance mismatch of the bar and the specimen. The rest is transmitted into the specimen, propagates through it, and reaches the second bar's interface with the specimen. At the second interface, part of the stress pulse is again reflected back into the specimen and the remainder is transmitted to the transmitter bar. If the specimen is much shorter than the wavelength of the loading pulse, the wave-transmitting time will be small compared with the duration of the loading stress pulse, and after a few reflections within the specimen, the stress and strain along the specimen become approximately uniform.

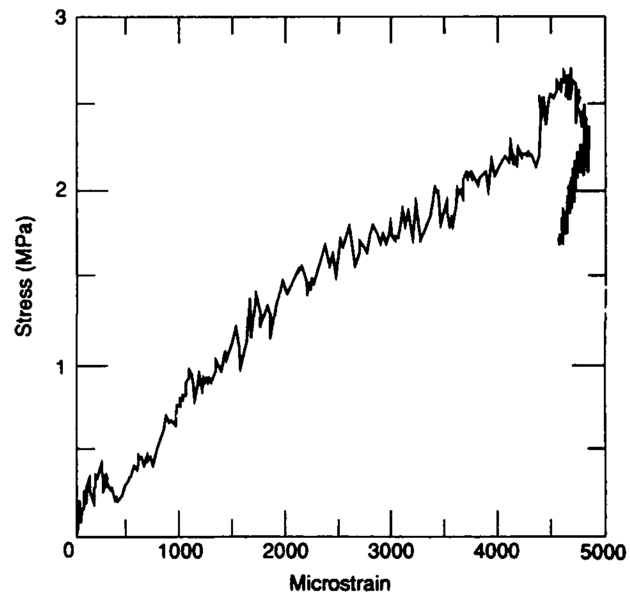
Consideration of equilibrium at the interface between the specimen and the transmitter bar shows that the forces in the specimen and in the transmitter bar are equal. The force is determined by measuring the strain on the elastic transmitter bar using strain gauges and the average strain in the specimen is calculated from the displacements at the end of the specimen. Figure 18 illustrates the high-speed (2-MHz sampling rate) data acquisition system using a 12-bit digital oscilloscope, which, with software developed at CRREL, can reconstruct the waveforms on an IBM PC with time shift for



a. Strain waveform records from the Hopkinson bars.



b. Computed strain rate from records in Figure 19a.



c. Computed stress–strain curve from the records in Figure 19a.

Figure 19. High-strain-rate fracture strain data from polycrystalline ice.

the above analysis. Figure 19 shows some data from high-strain-rate fracturing of polycrystalline ice samples.

Low-temperature ballistic test apparatus

Ballistic tests characterize not only a material's high-strain-rate fracture response to an impact by a relatively short impactor, but also the nature of damage the material sustains. Depending upon the velocity, the impactor penetrates either into or through the target material,

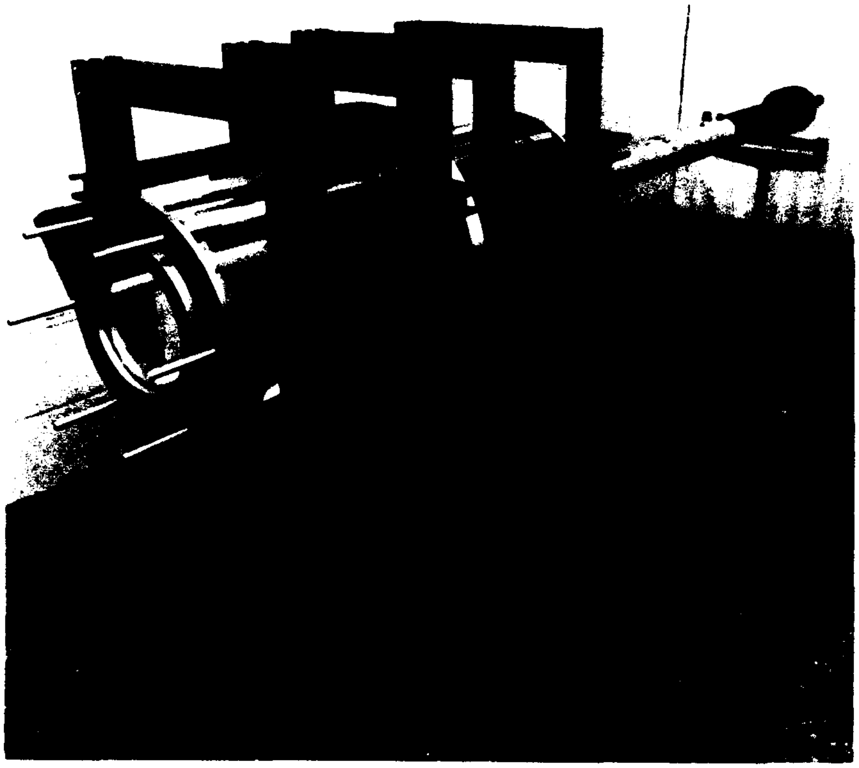


Figure 20. Low-temperature ballistic test apparatus.

inducing damage that is characterized as brittle–elastic, elasto–plastic, visco–elastic, plastic or completely hydrodynamic, the last indicating an extremely high velocity impact, in reaction to which the target material deforms as if it was a fluid. Again, for the same material, low temperature can drastically alter the nature of such fracture processes—a material may experience ductile deformation with small-area perforation damage at normal temperatures, whereas it experiences extensive brittle cracking over a large area at low temperature.

CRREL's low-temperature ballistic test apparatus (Fig. 20) is capable of firing projectiles of different shapes at high velocities (300 to 1500 m/s) at a target housed inside in a coldroom. Two chronograph screens at known distances apart, positioned just before the specimen target panel, and two immediately behind, give data on the impact and exit velocities of the impactor. The system has the option of establishing a vacu ım in the path of the projectile to avoid air turbulence and air shock to the target. In the vacuum system, the test panels are clamped in a sealed cylindrical housing of clear lucite for visibility and to allow chronograph lights to precisely trigger the electronic timers. The velocity measurements give data on energy absorption, while visual and microscopic examination of the target reveals the nature of damage. The lowest temperature of the coldroom is –28°C. For special tests at extremely low temperatures, a liquid nitrogen cooling system can be used.

Special support equipment

Scattered throughout various laboratories within CRREL, there are numerous other support equipment and apparatuses available to conduct in-depth study of any material A few are an acoustic emission testing apparatus (Fig. 21), a non-destructive testing apparatus and a very useful Scanning Electron Microscope (SEM). The last, a Hitachi model 500S SEM (Fig. 22), has a wide magnification range (×35 to ×100,000) and a good depth of field to allow study and photographs of surface features of different scales. Auxiliary to the SEM is a Kevex energy dispersive X-Ray detector that provides qualitative elemental analysis. The cold-stage attachment is an important feature of the SEM for performing microstructural analyses in the cold.

Another special and useful device for the study of the durability of materials at low temperatures is a thermal cycling apparatus, the Delta Design model 9064 temperature chamber (Fig. 23). With microprocessor-based solid state electronic controls and a computer interface, the apparatus is a stand-alone system for thermal cycling tests of materials in temperature ranges of 315 to –184°C. The internal air space volume of 62.3 L can be

Figure 21. Acoustic emission testing apparatus.

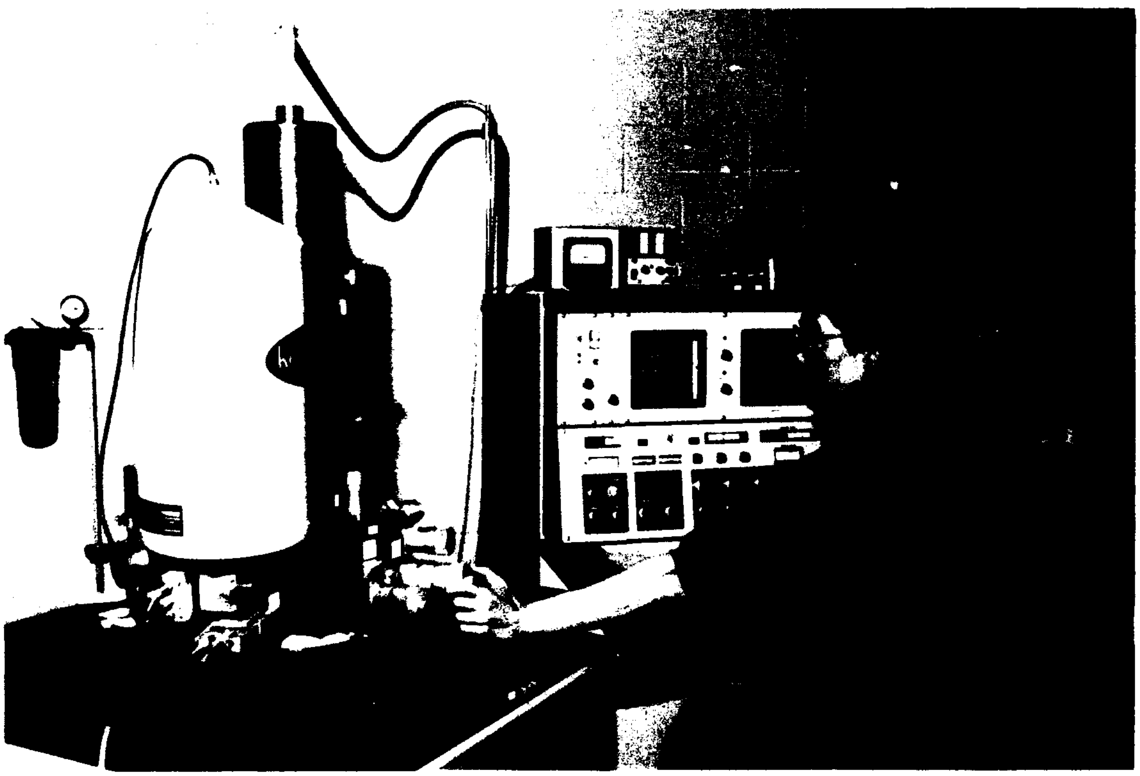


Figure 22. Scanning electron microscope laboratory.

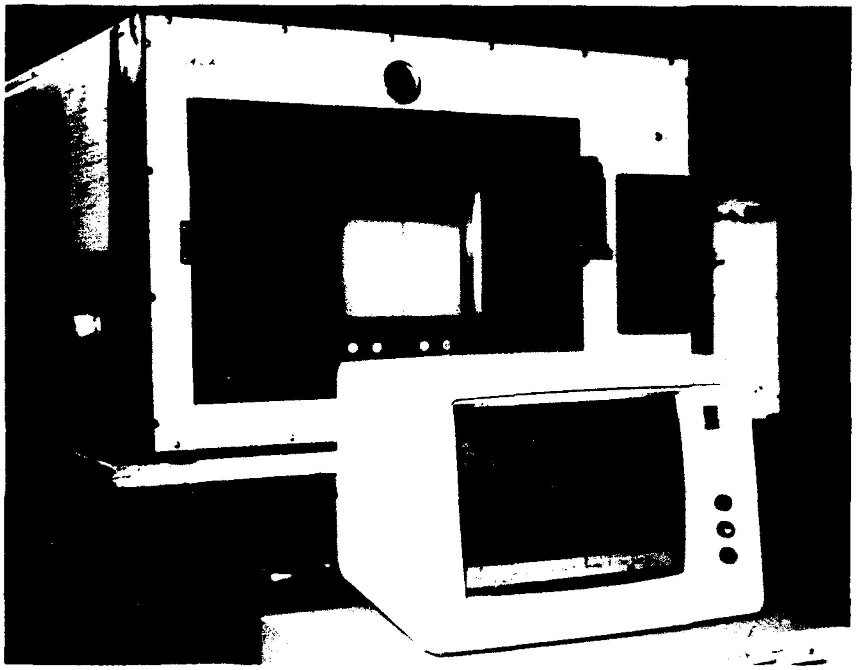


Figure 23. Computer controlled thermal cycling chamber.

heated up to 217°C by its internal nichrome heating elements in 28 minutes, while an external supply of liquid nitrogen, injected into the air stream through an expansion nozzle, can cool the chamber down to –53°C in a mere 4.5 minutes. Using specially developed software, a dedicated PC provides the automatic sequencing, rate of temperature change, soak time (time at a specified temperature), temperature set points, deviation limits and the number of passes for a run. A thermocouple system connected to a strip chart recorder keeps an alternative record of the chamber's temperature excursions.

Impedance matched shock gauge

Study of the dynamic behavior of a material requires a precise measurement of instantaneous stress within its interior. Insertion of gauges to sense this stress usually results in changing the material's impedance, a characteristic that controls its dynamic response. To overcome this problem, a new material, polyvinylidene fluoride (PVDF), a pressure sensitive polymer, has been evaluated to build gauges. A large number of gauges were built and evaluated on the Hopkinson Pressure Bar Apparatus, in which calibrated stress wave signals were passed and responses of the stress gauge were measured. Following the evaluation using various cladding materials, a design of the impedance matched shock gauge was developed. Figure 24 shows one such gauge.

Figure 24. Impedance matched shock gauge.

CONCLUDING REMARKS

Understanding the behavior of materials in the cold is the driving force behind the development of an organized materials laboratory at CRREL, in which facilities are being created to study both strain-rate and temperature influences. As mentioned before, one cannot be studied in isolation from the other. The research approach and development of the laboratory have considered both.

As a research material, composites have been the focus. In recent years, if there is any material that has promised revolutionary changes in human endeavors, it is composites. From space ship to battle ship, oil tank to army tank, composites are the material of choice, but low-temperature studies of this material are lacking. CRREL's materials research program is an organized effort to fill that void.

Degradation of composites under thermal cycling is well known, so is the development of residual internal stresses at low temperatures; however, the decrease in strength for unidirectional laminates was unexpected. A theoretical underpinning for this has been based on the wavy fiber theory. However, much more data, when available, are necessary to establish the validity of this theory more solidly.

Being a multidisciplinary research center, testing facilities and expertise resources are abundant within CRREL. A need for specialized equipment, especially devices for high-strain-rate testing, originally lacking, has now been met. With our focus on scientific and engineering problems encountered in cold regions, the additional facilities developed are specially adaptable for low-temperature studies.

In arctic and polar environments, where cold dominates all human activities, materials present a multitude of problems. There, in the cold, whether it is structural steel that becomes brittle, or composites that experience dramatically increased residual stress, or rubber seals or plastics that stiffen up, often with disastrous consequences, material behavior becomes a critical engineering issue. Engineering solutions are needed to these problems. CRREL's materials research is a necessary effort to address those critical problems.

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This report is a synopsis of the developments in the materials research program at CRREL. Focusing on studies of the low-temperature behavior of materials, the report reviews these developments in three specific areas: creating a materials-property data base, researching composites and other materials and developing test facilities. Among materials, composites, being newer, have been studied at depth. Temperature and strain rate have been considered critical in influencing any material's durability; therefore, facilities were developed to provide precise control of these parameters. The program aims to meet the crucial need for designing structures and equipment using materials specifically adapted for cold regions and low-temperature applications.

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